NPS ARCHIVE
1965
LEIBOVICH, H.

MAGNETIC DOMAINS AND TWINNING IN ALPHA-IRON

HARRY A. LEIBOVICH

U.S. NAVAL POSTGRADUATE SCHOOL
MONTEREY, CALIFORNIA

MAGNETIC DOMAINS

AND

TWINNING IN ALPHA-IRON

* * * * *

Harry A. Leibovich

MAGNETIC DOMAINS

AND

TWINNING IN ALPHA-IRON

by

Harry A. Leibovich
//
Lieutenant, Argentine Navy

Submitted in partial fulfillment of
the requirements for the degree of

MASTER OF SCIENCE
IN
ELECTRICAL ENGINEERING

United States Naval Postgraduate School
Monterey, California

1 9 6 5

NPS HERBARIUM
1965
LEIBOVICH, H.

Library
U. S. Naval Postgraduate School
Monterey, California

MAGNETIC DOMAINS

AND

TWINNING IN ALPHA-IRON

by

Harry A. Leibovich

This work is accepted as fulfilling

the thesis requirements for the degree of

MASTER OF SCIENCE

IN

ELECTRICAL ENGINEERING

from the

United States Naval Postgraduate School

ABSTRACT

Alpha-iron is another instance of mimetic twinning in which an edifice formed by a group of twinned tetragonal crystals presents an appearance of cubic symmetry. This confirms earlier suggestions that magnetic domains are crystallographic twins.

Crystals of alpha-iron were grown in thin strips of Armco iron, a large magnetic domain size confirmed by examining the Bitter pattern, the crystals examined point by point by means of Laue patterns and an oscillating crystal pattern of a crystal recorded. The Laue patterns confirm the persistence of a single crystal but shifts in relative intensities within the patterns show that clusters of differently oriented twins are illuminated by the x-ray beam at each point. The Laue pattern of a crystal shows a redistribution of relative intensities upon the application of an external magnetic field, indicating a new group of twinned orientations within the region illuminated by the x-ray beam. The pattern from a crystal, supposedly oscillating about a cubic [100] direction, shows layer lines which fit a twin lattice with a triple unit cell and twinned tetragonal crystals oscillating about [110] , [001] and [100] directions.

ACKNOWLEDGEMENT

The author would like to express his appreciation for the guidance and encouragement given him by Professor John R. Clark of the Material Science and Chemistry Department of the U. S. Naval Postgraduate School, throughout the course of this investigation.

TABLE OF CONTENTS

LIST OF ILLUSTRATIONS

LIST OF TABLES

1. Introduction.

A thorough search of existing literature indicates that the structure of iron has been determined only through powder studies. All of these determinations agree that the lattice is body-centered cubic. While it is difficult to be sure that the entire literature has been reviewed all of the standard references such as Donnay,[1] Wyckoff,[2] and Dana,[3] indicate only a cubic structure. The original literature reviewed is listed in the bibliography [1] to [11]. It is somewhat surprising that a more rigorous structural analysis has not been made since it is well known that the powder method does not indicate the presence of twins and is particularly susceptible to error in structures with mimetic twinning.

* * * * * * * * *

[1] Donnay, J. D. H Crystal data. Determinative tables (1963).

[2] Wyckoff, R. W. G. Crystal structures (1948).

[3] Dana, J. D. System of Mineralogy (1944).

CHAPTER I

FAULTING AND MAGNETIC THEORY

1. Face-centered tetragonal description of body-centered cubic lattice.

It is possible to describe the body-centered cubic lattice in terms of a face-centered tetragonal cell with an axial ratio of $c/a = \frac{1}{2}\sqrt{2}$. In the same coordinates a face-centered cubic lattice is a face-centered tetragonal lattice with an axial ratio of unity. Comparing the two structures in these coordinates, the (111) plane is the close-packed or pseudo-close-packed plane, and faulting takes place in both systems on this plane. In the face-centered cubic system all directions in the form $[112]$ or in the form $[110]$ are equivalent and twenty-four twinned orientations are possible. In the tetragonal coordinates the directions of the form $[110]$ are no longer necessarily all equivalent, nor are the directions of the form $[112]$. This can be understood when one considers that in the face-centered cubic, a close-packed structure, the (111) planes are also close-packed as shown in figure 2. In the body-centered cubic structure the "close-packed" planes are only pseudo-close-packed as shown in figure 1. Because, as tetragonal coordinates make clear one of the $[112]$ directions is unique, there are only eight twin orientations.

The axial ratio in a tetragonal crystal determines the strain energy associated with a dislocation, since the strain energy is

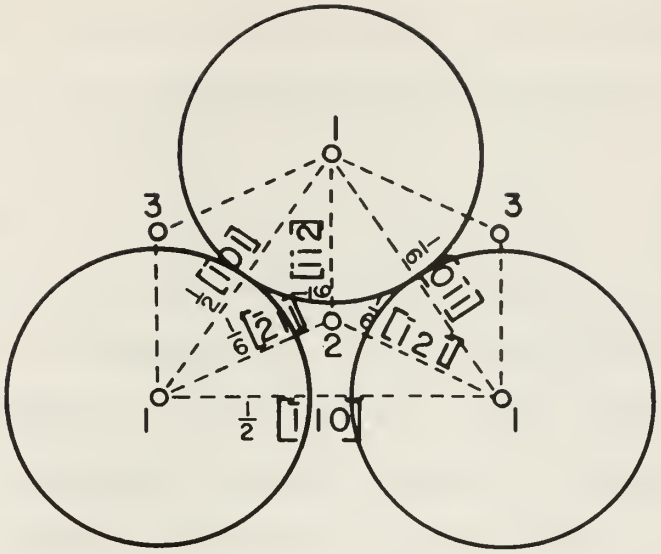

FIGURE 1

FACE CENTERED TETRAGONAL

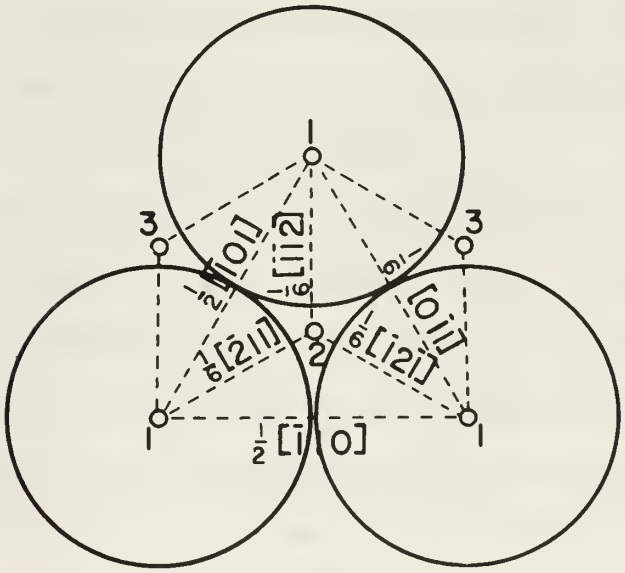

FIGURE 2

FACE CENTERED CUBIC

proportional to the square of the Burguer's vector. The axial ratio also determines the direction in which dislocation reactions involving partial dislocation and the development of stacking faults will proceed.

2. Twins

Since the cubic lattice can be described as tetragonal it is particularly appropriate to do so in discussing iron because iron is ferromagnetic. Any crystal with a polar moment cannot have a center of symmetry; therefore, although the tetragonality may be slight, symmetry does require that the crystal be tetragonal. It has been recognized for many years that tetragonal crystals with an axial ratio close to unity form a twinned structure which possesses a pseudo-cubic symmetry. This mimicing of a symmetry, higher or lower than that of its constituents, by a twinned edifice is known as mimetic twinning, and is described in crystallographic texts such as by Phillips.[1]

Figures 3 and 4 illustrate the construction of lattices by stacking successively (111) planes. Figure 3 shows the well known face-centered cubic lattice based on this layer structure. Figure 4 shows that by stacking at an angle to the (111) plane a body-centered cubic structure can also be constructed, with $c/a = \frac{1}{2}\sqrt{2}$. With $c/a = 1$ the direction $[111]$ is normal to the

---

[1] Phillips, F. C. An Introduction to Crystallography (1955).

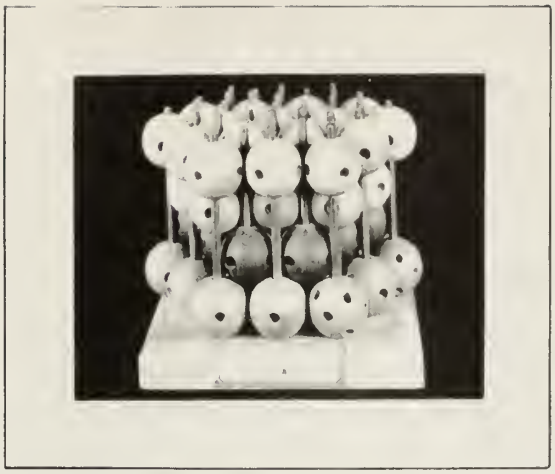

FIGURE 3

STACKING OF (111) PLANES IN
A FACE-CENTERED CUBIC LATTICE

FIGURE 4

STACKING OF (111) PLANES IN
A BODY-CENTERED CUBIC LATTICE

planes (111); when $c/a = \frac{1}{2}\sqrt{2}$ the $[111]$ direction makes an angle of 71.5 with the (111) planes. In both structures a shear of $1/6[\bar{1}\bar{1}2]$ produces a stacking fault. This faulting is well known and frequently described in connection with the face-centered cubic lattice as the sequence A, B, C, B, C, ... , produces a shear, or, upon reversal of the stacking sequence after faulting, resulting in a sequence A, B, C, B, A, ... , producing a twin. The occurance of a fault by a $[\bar{1}\bar{1}2]$ shear on the (111) plane, is analogous in the face-centered tetragonal lattice with $c/a = 1$ and $c/a = \frac{1}{2}\sqrt{2}$.

From this point of view it is possible to describe the edifice resulting from a twinning shear in the face-centered tetragonal lattice. This twinned edifice will be a structure resulting from combinations of the eight possible twinned orientations. As each of the possible shears operate, a new twinned orientation is added to the edifice; when, finally, all eight (or possibly, only four,) of the twin orientations have been developed the twinned edifice shows a cubic symmetry which, of course, is more properly described as pseudo-cubic.

The geometrical laws of twinning require that a twinned edifice contain a crystallographic symmetry element which is not a symmetry element of the individual crystals; they also require that the twin edifice possess a lattice with a unit cell which is a multiple, or sub-multiple, of the unit cell of the individual crystals.

In face-centered cubic or face-centered tetragonal crystals the unit cell of the twinned lattice is three times the dimension of the unit cell of the individual crystal in each direction. The unit of the twinned edifice, therefore, contains twenty-seven of the original unit cells and 108 atoms. The coordinates of the atoms in the original edifice are multiples of unit translations of the four lattice vectors of the face-centered tetragonal lattice.

The coordinates of atoms in the twinned edifice are obtained by applying a shear to the original atoms in the appropriate $[112]$ direction on each of the planes of the form (111). The composite structure thus obtained resembles a space lattice but must be differentiated from a space lattice. Each of the points in this "lattice" is occupied by atoms of an individual crystal of the twin array. Some, but not all, of the coordinates are occupied by atoms in more than one crystal, i.e., are common to more than one twin orientations.

3. Magnetic Theory - Domains

The fact that iron crystals are tetragonal was pointed out in 1937 by Jaffe[2] in a discussion of the structure of a ferroelectric crystal, Rochelle Salt. He notes:

> It is possible to extend the crystallographic argument of this paper to the ferromagnetic crystals themselves

[2]Jaffe, Hans Von R. Physics Review 51, 43 (1937).

> and to postulate that an iron single crystal is a pseudo-cubic crystal; and that the real crystal individuals are the Weiss regions, which have, in absence of an applied magnetic field, a spontaneous polarization in the direction of one of the cubic edges and are therefore tetragonal paramorphic.

He adds:

> This point of view seems correct, but hardly fertile, ...

The point of view is still correct and because of the development of dislocation theory in the intervening years many consequences are more apparent today than they were in 1937. Jaffe's conclusions concerning the twinned structure of Rochelle Salt have long since been proven correct. However, no investigations of the structure of iron in accordance with his suggestions has been reported. The extensive studies of Bozorth[3], Shockley[4], Williams[5], and their co-workers have given visible evidence of the domains in ferromagnetic materials, but neither their work nor the work of Kittel[6], Galt[7], and others on domain wall movements has linked domain structures to crystallographic features of the crystals.

[3]Bozorth, R. M. Ferromagnetism, (1961).

[4]Shockley, W. Dynamic experiments with a simple domain boundary, (1951).

[5]Williams, H. J. Elec. Eng. 69, 817-822 (1950). Ferromagnetic domains.

[6]Kittel, C. Introduction to Solid State Physics (1956).

[7]Galt, J. K. and Kittel, C. Solid State Physics 3, (1956).

Apparently, because of the close analogy between ferroelectricity and ferromagnetism some workers, as for example, Klassen and Neklyudova[8], take for granted that domains are mechanical twins. No evidence heretofore has been adduced to establish this.

---

[8] Klassen and Neklyudova. Mechanical twinning of crystals (1964).

CHAPTER II

PREPARATION OF SPECIMENS

1. Growth of single crystals of iron by the strain - anneal method

No information seems available concerning methods by which the size of twins may be controled but comparison of microstructures indicate that, in general, the twins found in fine grain materials are small. Therefore, since we wish to examine the crystal structure of what may be twinned, it is desired that the crystals be as large as possible.

In iron, coarse grains must be grown by the strain anneal method because of the alpha-gamma transformation.

Large crystals of alpha iron were grown after the following procedure was developed by trial and error.

a. Armco iron was cold rolled from one inch square bars to form to strips about 10" x 1" x 1/32".

b. The cold rolled strips were annealed in a hydrogen atmosphere for forty-eight hours at 920°C, then cooled to 100°C in twelve hours. Grain sizes of approximately 120 grains per square millimeters were obtained.

c. The annealed strips were elongated 3.25 per cent in a tensile testing machine.

d. The strained strips were annealed seventy-two hours at

870°C in a hydrogen atmosphere and cooled to room temperature in five hours.

With this procedure it was possible to obtain single crystals with dimensions of about 1" x ½".

2. Bitter Patterns

The structure of magnetic domains is revealed by the application of a ferromagnetic colloid as described by Elmore[1], which under a microscope is seen to concentrate at the domain boundaries. The domain structure of the large crystals prepared for study is seen in figure 5 in which the domains are delineated in a field of 1600 oersteds. The Bitter patterns show that the domains are approximately one millimeter wide and a change in orientation at the grain boundaries is apparent.

In order to observe Bitter patterns the specimen must be carefully prepared and disturbed material on the surface removed by electropolishing. Some difficulty was experienced in finding a suitable electrolite to polish these specimens. The electrolite used was prepared as follows: 75 grams of Ferric Nitrate and 75 grams of Manganese Nitrate, dissolved in 450 cubic centimeters of Methanol. Dissolve 3 grams of Urea and 15 grams of Citric Acid in Distilled water. Add the second mixture to the first, next, 450 cubic centimeters of Ethanol and 30 cubic centimeters of Ethanol and 30 cubic centimeters of Nitric Acid (70 per cent, S. G. 1.67). Boil in a water bath until it reaches the boiling point.

[1]Elmore, W. C. Phys. Rev 54, 309 (1939).

FIGURE. 5

MAGNETIC DOMAINS STRUCTURE

Cool at room temperature and let the precipitate dissolve for twenty-four hours. Apply 1.25 amperes for ten seconds placing the specimen in a beaker.

During observation of the Bitter patterns under the microscope it was found that the colloidal solution evaporated quickly in the heat from the solenoid providing the field. It was found that this evaporation could be materially slowed by the addition of 10 per cent of glycerin to the colloidal solution.

CHAPTER III

X RAY EXAMINATION

1. Laue Patterns

   a. Patterns at successive points across the crystal

   Having insured that the crystals grown had large magnetic domains, comparable to the size of the X ray beam, Laue patterns were taken at a series of points, separated by one or two millimeters, across the grains. The specimen was mounted and oriented in a goniometer head and translated by means of the horizontal guide in a [001] direction between successive photographs. The patterns obtained are shown in figure 6.

   Analysis of these patterns shows a very wide range of relative intensities in the diffracted beams recorded in the pattern. Although there is no change in the orientation of the crystal, demonstrated by the same patterns of diffracted beams, the relative intensities among the diffracted beams vary from point to point in the single crystal. This shift in relative intensities of diffracted beams is significant because, ordinarily, the difference in intensity of diffracted beams in Laue patterns is attributed to the varying intensities of the wave lengths in the emission spectrum picked off by each diffrac-

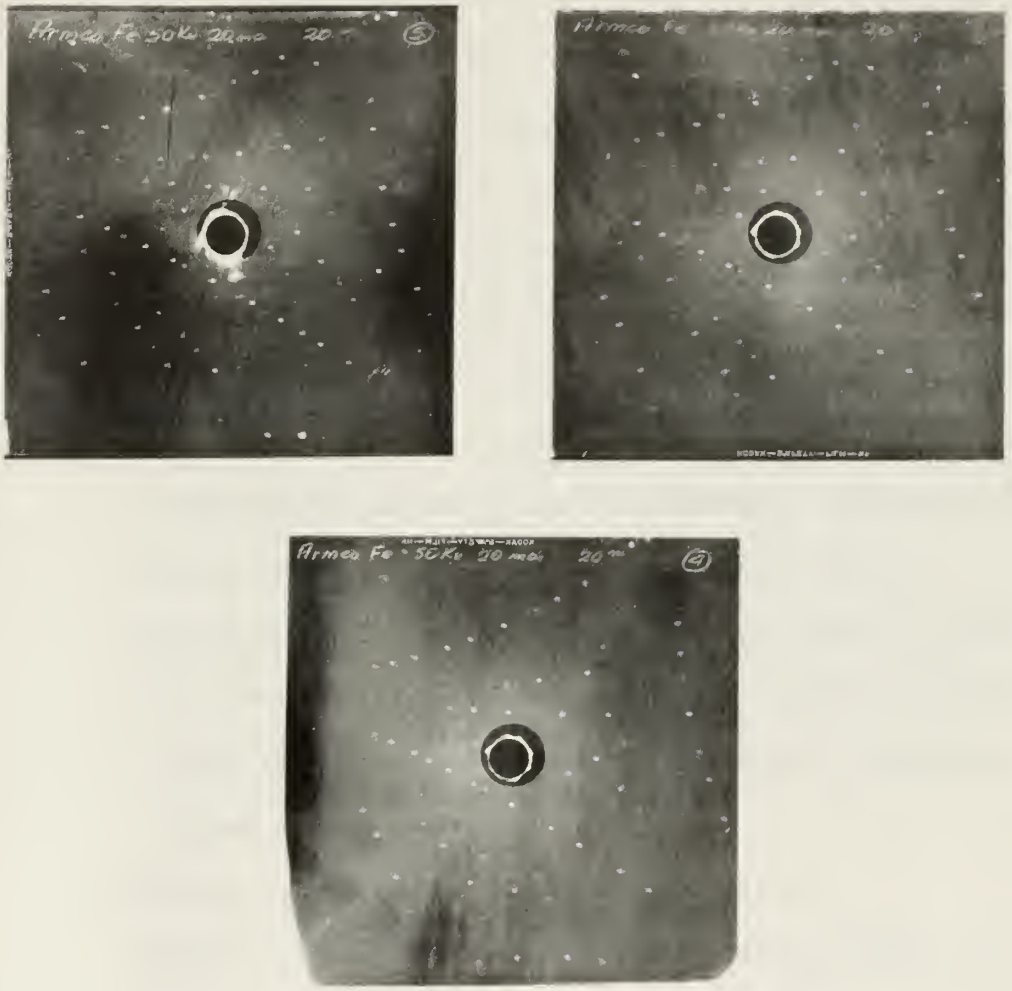

FIGURE 6

LAUE PATTERNS AT SUCCESSIVE POINTS ACROSS THE CRYSTAL

ting plane. However, it should be noted that the difference in intensity in the spectrum of white radiation from a tube operating at about 50 kilo volts is of the order of three or four to one over the range of wave lengths from the short wave length cut-off to the longest wave length diffracted by the crystal.

The other factors which can account for these differences in relative intensities, are the angular factors, and the the structure factors of the diffracting planes. Since the structure factor for all planes in a cubic or a tetragonal crystal is the same, the differing structure factor must be those of the plane of the twinned edifice. Microphotometer traces of corresponding points in figure 6 are shown in figure 7, indicating the relative intensities of the same same pairs of diffracted beams in different regions of the crystal.

b. Effect of a Magnetic Field on Laue Patterns

Additional evidence of the relation between domain and crystallographic orientations is the reorientation of domains effected by the application of a magnetic field. In figure 8a a diffraction pattern obtained without a magnetic field is shown. Figure 8b shows a diffraction pattern from the same area with field of 1200 oersteds applied to the specimen. Figure 8c shows a third diffrac-

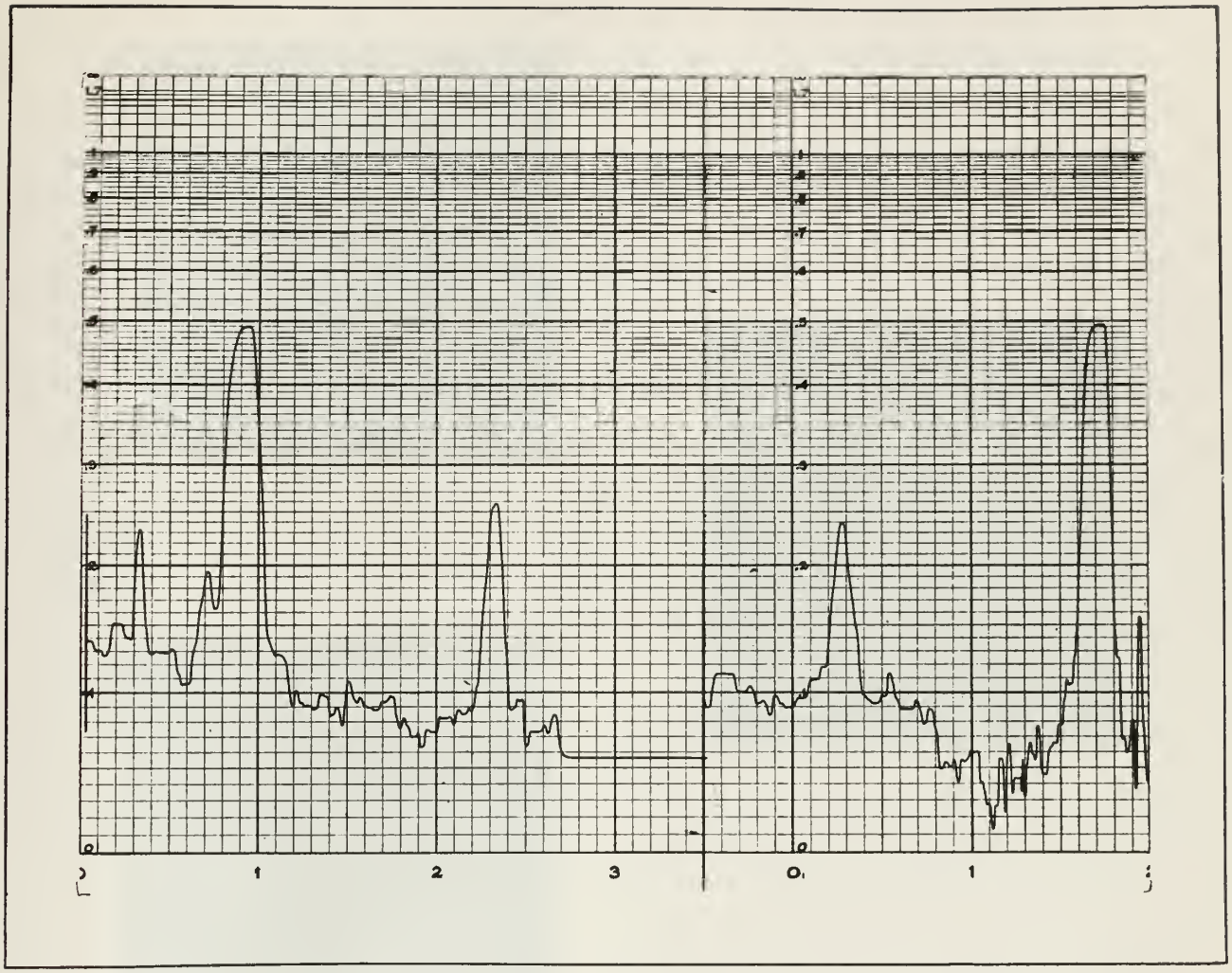

FIGURE 7

MICROPHOTOMETER TRACES OF THE SAME PAIRS OF DIFFRACTED BEAMS IN DIFFERENT REGIONS OF THE CRYSTAL

FIGURE B

FIGURE

tion pattern, the crystal still untouched, but with the field reversed. The variation in the relative intensities of the same pairs of spots shows that the twin orientations present in the region illuminated by the X ray beam have been altered by the application of the magnetic field.

2. Oscillation Crystal Patterns

It had been intended to record rotation patterns from relatively small crystals cut from large crystals grown in the rolled sheet. However, it was noted when the smaller crystals were being cut with a jeweler's saw from the large crystals, the grain boundaries moved in response to the disturbance caused either by the sawing or the image forces produced by introducing a new surface into the network of dislocations. For this reason the long strip containing the crystal was mounted in a somewhat awkward arrangement and the diffraction pattern limited by the range over which the strip could be oscillated without mechanical interferences. A selected crystal was oriented by means of Laue patterns so that [001] direction was perpendicular to the axis of oscillation. Oscillating patterns were then taken at 35 kilo volts and 20 milliamperes, six hour exposure, Zr filter to remove the beta radiation from Mo K radiation. The crystal was oscillated through an angle of 43° and produced the pattern shown in figure 9 recorded on a flat film 5 centimeters from the crystal. The pattern was measured with a Bernal Chart and the layer

FIGURE. 9
OSCILLATION CRYSTAL PATTERN

line spacings are given in Table I.[1] This data can be interpreted on the basis of a structure containing crystals oriented to oscillate about the directions [001] , [100] and [110] . In the first direction the unit cell is oriented as shown in figure 10 and rotates around the [110] axis, which is the twinned direction of the [001] orientation. This unit cell has in the reciprocal lattice an interlayer spacing of

$$d^*_{twinned} = \frac{\lambda}{d_{[110]}} = \frac{\lambda}{3 \times 2 \times a} = 0.041$$

Where

λ: wave length for Mo K radiation (0.707 Å)

a: lattice parameter for Fe(2.866 Å)

The unit cell oriented in the [100] direction, as shown in figure 11 rotates around the [001] axis and it has in the reciprocal lattice an interlayer spacing of

$$d^*_{untwinned} = \frac{\lambda}{d_{[001]}} = \frac{\lambda}{3 \times \sqrt{2} \times a} = 0.058$$

The two tetragonal orientations can be regions in one cubic structure separated by Frank partial dislocations.

---

[1]Buerger, M. J. X Ray Crystallography, Wiley, (1942).

TABLE I: OSCILLATION PATTERN - LAYER LINE SPACING

Upper	L twinned	L untwinned
0.04	-	1
0.04		
0.12	2	3
0.12		
0.16	-	4
0.16		
0.16		
0.16		
0.24	4	6
0.24		
0.24		
0.24		
0.28	-	7
0.28		
0.32	-	8
0.32		
0.32		
0.32		
0.40	-	10
0.40		
0.48	8	12
0.48		
0.53	9	13
0.53		
0.53		
0.53		
0.57	-	14
0.57		
0.62	10	15
0.62		
0.62		

Lower	L twinned	L untwinned
0.06	-1	-
0.06		
0.12	-2	-3
0.12		
0.23	-4	-6
0.23		
0.28	-	-7
0.28		
0.28		
0.28		
0.32	-	-8
0.32		
0.32		
0.32		
0.38	-6	-
0.38		
0.49	-8	-12
0.49		
0.58	-10	-14
0.58		
0.58		
0.58		
0.63	-	-15
0.63		

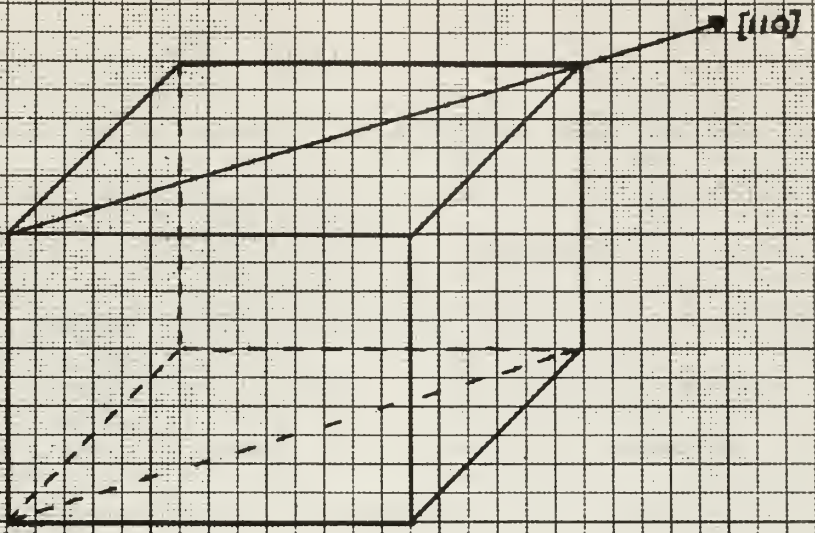

FIGURE 10

ROTATION AROUND [110]

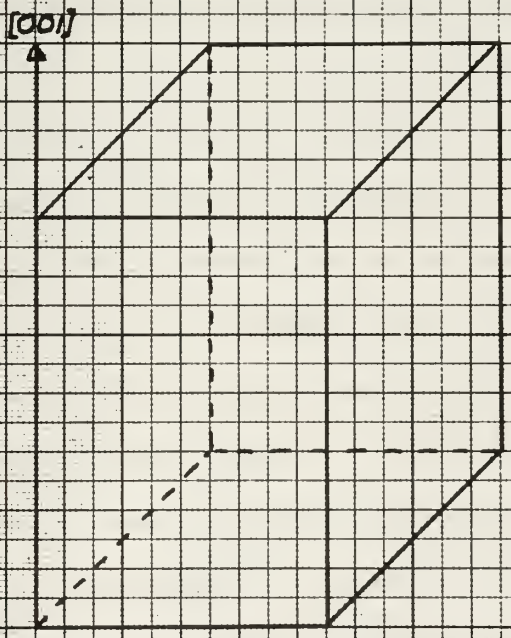

FIGURE 11

ROTATION AROUND [001]

CONCLUSIONS

1. Structure - Domains and Twins

The evidence obtained indicates that ferromagnetic domains are crystallographic twins within a twinned edifice. The symmetry of tetragonal crystals, therefore, allows the magnetic domains within the polydomain edifice to be oriented in eight different directions. Therefore iron is another example of mimetic twinning. The superlattice formed by the twinned crystal simulates the symmetry of a single crystal of higher symmetry than the actual crystals. The accepted data on the structure of iron, recording only body-centered cubic symmetry, results from the fact that determinations have been made only by powder patterns which do not differentiate between twinned and untwinned crystals. The oscillating crystal pattern shows clearly a large unit cell which coincides with the triple unit cell of a twin crystal of iron. The differences in intensities in the Laue patterns from different regions in the same crystal indicate that a different array of twins is present in the beam as it strikes the different points in the crystal. More strikingly the application of a magnetic field also produces a re-grouping of twinned orientations as evidenced by the re-distribution of intensities among the diffracted beams observed in the pattern.

2. Magnetic properties - Domain walls as dislocation walls

Stacking faults introduce twinned regions bounded by partial dislocations within a crystal. These dislocation walls require energy to be moved thus, the magnetic field applied to move a domain wall must not only supply the energy necessary to re-arrange the magnetic ordering i.e. spin coupling, but must also supply energy to overcome the elastic fields surrounding the dislocations. Thus, part of the energy required to move a domain wall is that necessary to overcome the Peierl's forces and to break the dislocations away from Cottrel atmospheres or other anchoring impurities. The existence of twinned regions in alpha-iron is also of major importance in determining the mechanical properties of iron. Twinning is a mode by which a crystal can be deformed; however, the existence of twin walls interferes with the movements of dislocations, causes dislocation pile-ups and is certainly associated with the transition temperature observed in iron and low carbon steel.

BIBLIOGRAPHY

1. Blake, F. C. Physics Review 26, 60 (1925).

2. Davey, W. P. Physics Review 25, 753-761 (1925).

3. Hull, W. Physics Review 9, 84 (1917). Physics Review 10, 661-696 (1917). Physics Review 14, 540 (1919).

4. Jette and Foote Strukturbericht III, 187 (1935). Strukturbericht II, 194 (1935).

5. McKeehan, L. W. Physics Review 21, 402-407 (1923).

6. Owen, G. D. and Preston, Proc. Physics Society of London 35, 101-108 (1923).

7. Owen, G. D. and Yates, Strukturbericht III, 182 (1933). Strukturbericht II, 164-195 (1933). Strukturbericht III, 346 (1933). Strukturbericht I, 16 (1933).

8. Thomas, J. Structure Reports XI, 137 (1948).

9. Westgren, A. Zeitschr. f. Phys. Chem. 98, 181-210 (1921). Engineering 111, 727-728, 757-759 (1921). Zeitschr. f. Phys. Chem 102, 1-25 (1922). Journal Iron and Steel Inst. 105, 241-273 (1922). 109, 159-174 (1924). Engineering 113, 630-634 (1922). Nature 94, 114 (1924)

10. Wever, F. Mitt. a. d. K. W. Institut f. Eisenforschung 8, 45-58 (1921). Naturwissenschaften 114, 1106-1114 (1924).

11. Wyckoff, R. W. G. and Crittenden, E. D. Journ. Am. Chem. Soc. 47, 2868-2876 (1925).

thesL475
Magnetic domains and twinning in alpha-i
3 2768 002 12035 4
DUDLEY KNOX LIBRARY